

# A New Test Chamber To Measure Material Emissions under Controlled Air Velocity

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A new 20-L glass chamber for the determination of VOC emissions from construction materials and consumer products under controlled air velocity and turbulence is described. Profiles of air velocity and turbulence, obtained with precisely positioned hot wire anemometric probes, show that the velocity field is homogeneous and that air velocity is tightly controlled by the fan rotation speed; this overcomes the problem of selecting representative positions to measure air velocity above a test specimen. First tests on material emissions show that the influence of air velocity on the emission rate of VOCs is negligible for sources limited by internal diffusion (e.g., PVC tile) and strong for sources limited by evaporation. In a velocity interval from 0.15 to 0.30 m s<sup>-1</sup>, an emission rate increase of 50% has been observed for pure *n*-decane and 1,4-dichlorobenzene and of 30% for 1,2-propanediol from a water-based paint. In contrast, no measurable influence of turbulence could be observed during vaporization of 1,4-dichlorobenzene within a 3-fold turbulence interval. Investigations still underway show that the chamber has a high recovery (i.e., low adsorption) for the heavier VOC (TXIB), even at low concentrations (~20 μg m<sup>-3</sup>).

## Introduction

Emission testing of materials in large environmental chambers was first performed for the determination of formaldehyde emitted from wood-based panels. Successive emissions of organic vapors (VOC) were also investigated by means of small test chambers: after the pioneering work by Møhlhøve (1), a more systematic approach was conducted by Tichenor et al. (2) with smaller and smaller test chambers until guidelines for the determination of VOC emissions were published (3–5).

In a validation effort of the European Guideline (5), two interlaboratory comparisons were organized on different materials; the results showed an acceptable interlaboratory agreement for the solid material tested (PVC tile) but unacceptable discrepancies for the two “wet” materials tested, a floor wax and a paint (6–8). The preparation of the wax and paint film specimens, analytical errors, differences in air velocity/turbulence, and vapor losses to the chamber walls (sink effect) were the main reasons pointed out for the bad results. The challenge of overcoming these difficulties by further research was accepted by a group of research laboratories and industrial companies (see the list in the Acknowledgment section), which undertook a cooperative effort with the aim of improving accuracy and precision in the determination of VOCs emitted from building materials

and products in small test chambers. In the frame of this European Project named VOCEM (VOC Emission), partially financed by the European Commission, the development of the new chamber described also took place.

The development of the new chamber was justified by the following reasons. There is a need for very compact, low-cost test chambers to face the increasing demand of long duration materials testing for chemical pollution (28 days according to ref 9 and even 26 weeks according to ref 10). The chamber must have a space with known and controlled air velocity/turbulence in a realistic range, i.e., 0–0.30 m/s for indoor spaces (offices, homes, schools, etc.); the internal recirculation rate and the air change rate with external air should be high to make attainment of steady-state concentrations faster and to reduce losses due to wall adsorption on emission measurements. At the same time, the area-specific ventilation rate (flow rate normalized to the material area) should match the values adopted for different materials (e.g., flooring, wallcovering, sealing) (9, 11). The chambers realized during the last years do not entirely meet these requirements. The FLEC (Field and Laboratory Emission Chamber), a stainless steel microchamber with 35 cm<sup>3</sup> capacity (12), has several interesting features, but it also presents drawbacks. The principal ones are that (a) the specimen area is too small to balance the lack of homogeneity of materials and (b) the air velocity can neither be measured with common anemometers nor varied independently from the air change rate. For the CLIMPAQ (Chamber for Laboratory Investigations of Material Pollution and Air Quality), a 50-L glass chamber designed for simultaneous chemical and sensory emission testing of materials (13), no data on air velocity/turbulence profiles are known. Two chambers with controlled air velocity were developed in Canada (14, 15), but they require a “jacket” chamber if emission measurements have to be carried out.

## Materials and Methods

The new chamber has two special features: (a) an axial fan used for air circulation and, as a consequence, (b) homogeneous, well-characterized, and adjustable conditions of air velocity and turbulence above the emitting material. Chamber design and construction was achieved in two steps. First, an experimental setup was realized including the following parts: (a) a glass box (30 cm width, 50 cm length, 10 cm height) wherein air velocity and turbulence could be controlled: the two smaller sides (air inlet and outlet) were open, a PTFE (polytetrafluoroethylene) grid being installed on the inlet; (b) a much larger glass chamber (450 L) into which the box was installed and through which air was recirculated; (c) an axial fan, i.e., a fan with (aluminum) blades parallel to the shaft, installed at one end of the box through which it sucked the air, the fan was driven by a dc motor, with dynamometric control, ensuring a precise, continuously adjustable rotation speed between 0 and 1800 rpm; (d) a surface gauge for the precise positioning (0.1 mm) of the anemometric probe, this device has been motorized to speed up the operation.

The final chamber (see scheme in Figure 1) has been realized with 10-mm glass panes glued together with Loctite. The axial fan is driven by a compact dc motor with dynamometric control and continuously adjustable speed between 0 and 1000 rpm; however, the air motion starts to be measurable by the anemometer at 300 rpm. To reduce adsorption/desorption problems, the motor and the bearings of the shaft are mounted outside the chamber, and PTFE joints are used as seals between shaft and chamber. To

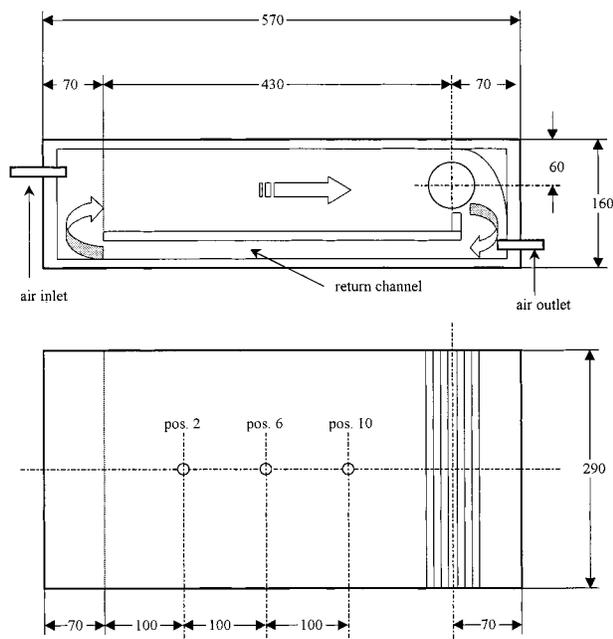


FIGURE 1. Scheme of the new chamber with positions of velocity/turbulence profiles.

TABLE 1. Main Physical Characteristics of the New Chamber

parameter	value
sizes (external length, width, height) [cm]	57, 29, 16
volume [L]	20.8
area for the test material (max) [m <sup>2</sup> ]	0.35
loading ratio (max) [m <sup>2</sup> m <sup>-3</sup> ]	16.8
air flow rate (max) [L min <sup>-1</sup> ]	14
air change rate (max) [h <sup>-1</sup> ]	42
area specific ventilation rate [m <sup>3</sup> h <sup>-1</sup> m <sup>-2</sup> ]	2.4 <sup>a</sup>
internal mixing rate (300–700 rpm) [h <sup>-1</sup> ]	396–924
air velocity [m s <sup>-1</sup> ]	0.08–0.40

<sup>a</sup> At maximum loading and air exchange rate.

facilitate cleaning of the chamber, the fan is easily detachable. The chamber is accessible by removing the cover plate, which is sealed by a PTFE gasket. The main physical features of the chamber are summarized in Table 1.

The fan sucks the air over the specimen and recirculates it through a return channel: the recirculation rate is >400 h<sup>-1</sup> (396 h<sup>-1</sup> at the minimum speed of 300 rpm or 0.088 m s<sup>-1</sup> above the boundary layer). This internal mixing rate is 1 order of magnitude greater than the highest exchange rate of chamber air with external air. A stainless steel wire gauze (1600 mesh cm<sup>-2</sup>), on the side opposite to the fan, defines the space with controlled air velocity/turbulence: this gauze filters eddies (i.e., streamlines the airflow) much more efficiently than the PTFE grid used in the preliminary setup. The specimen area can be varied: from one to four 875 cm<sup>2</sup> (27 cm × 32 cm) modules; they can be accommodated on the bottom, on the top (by means of screws), and on both sides of an intermediate support. The maximum total specimen area of 3500 cm<sup>2</sup> corresponds to a loading ratio of 16.8 m<sup>2</sup> m<sup>-3</sup>. Apart from increasing the analytical sensitivity, a higher loading factor is desirable to decrease the losses due to sinks, i.e., deposition of vapors on the internal chamber surfaces. The airflow rate through the chamber can be varied up to 14 L/min (42 h<sup>-1</sup>). The air inlet and outlet are realized with Swagelok connections; a Tee piece is installed on the outlet to enable sampling of the chamber air. The chamber is equipped with two cover plates: one with holes for anemometric probes is installed only for velocity/turbulence

characterizations; the other leak-tight one is employed for all chemical measurements.

The airflow pattern inside the box and the chamber has been studied by means of a StreamLine constant temperature hot wire anemometer (manufactured by Dantec, Skovlunde, Denmark). This instrument is an integrated measuring system based on a hot wire probe, a thermocouple, a constant temperature module, a PC, and especially developed software. The system is designed to maintain constantly the temperature of the wire, counteracting the cooling effect of the airflow being measured: the anemometric signal is given by the voltage required to keep the wire temperature constant. For each measurement, the instrument can give the mean air velocity and the associated turbulence (root mean square, RMS, i.e., the standard deviation) over a selected time interval and with a selected signal sampling frequency. The instantaneous velocity can also be visualized. The turbulence can be expressed in meters per second and also in percent, if it is divided by the mean velocity and multiplied by 100, like it is usually done for the standard deviation. The calibration of the hot wire probes is performed by means of an automatic calibration system (also by Dantec) in the range between the detection limit and 0.5 m s<sup>-1</sup>. The instrument is characterized by the following performances: (a) the detection limit is 0.03 m s<sup>-1</sup>; (b) the accuracy is given by the (frequent) calibration of the probe through the system above (see precision below); (c) the precision of the probe (inclusive of the turbulence of the airflow generated by the calibration system, as the two parameters cannot be distinguished) is ±1%. The duration of the measurements has been 0.5 s (frequency of 1000 Hz signal points acquisition) for the new chamber. For the old type chamber, tests have been carried out with two air velocity averaging times, 0.5 and 60 s: the results showed differences between 12% (at 0.3 m s<sup>-1</sup>) and 47% (at 0.06 m s<sup>-1</sup>). This is additional evidence of the uncertainty in measuring air velocity within this type of chamber.

The thermostatzation of the chamber and regulation of RH therein have been already described elsewhere for another 0.45 m<sup>3</sup> chamber (16). The airflow rate through the chamber is controlled by a mass flow controller (Brooks Instrument B.V., Veenendaal, The Netherlands), the calibration of which is checked by an electronic flowmeter based on the displacement of a piston (primary standard, model Dry-Cal DC-1, Bios International Corp., Pompton Plains, USA). Accuracy and precision of flow rate measurements are estimated to be within ±3%.

The data on temperature, relative humidity, and airflow rate are acquired every 10 min on a data logger (model 500, Data Electronics, Letchworth Garden City, Hertshire, U.K.). Twice a month, the data collected are transferred to a PC, where they are imported in a MS EXCEL spread sheet; a graph is produced along with values of mean, standard deviation, minimum, and maximum for each parameter.

VOC (C6–C16) concentrations are determined by sampling chamber air (usually 1 L) on Tenax TA cartridges that are thermally desorbed at 250 °C and injected into a GC-FID apparatus equipped with a OV-1 capillary column. The identification of compounds is carried out by GC-MS analysis, using a HP 5972 mass spectrometer with automatic library search. Details of the analytical procedures are described elsewhere (17).

## Results and Discussion

**Characterization of Air Velocity and Turbulence.** Much work was performed to characterize the air movement inside the controlled space in the preliminary setup (see Supporting Information). Vertical profiles of air velocity and turbulence, obtained by measuring at tight height intervals (1 mm in the boundary layer, i.e., the layer where air velocity presents the gradient from zero to the maximum value, and 5 mm upward)

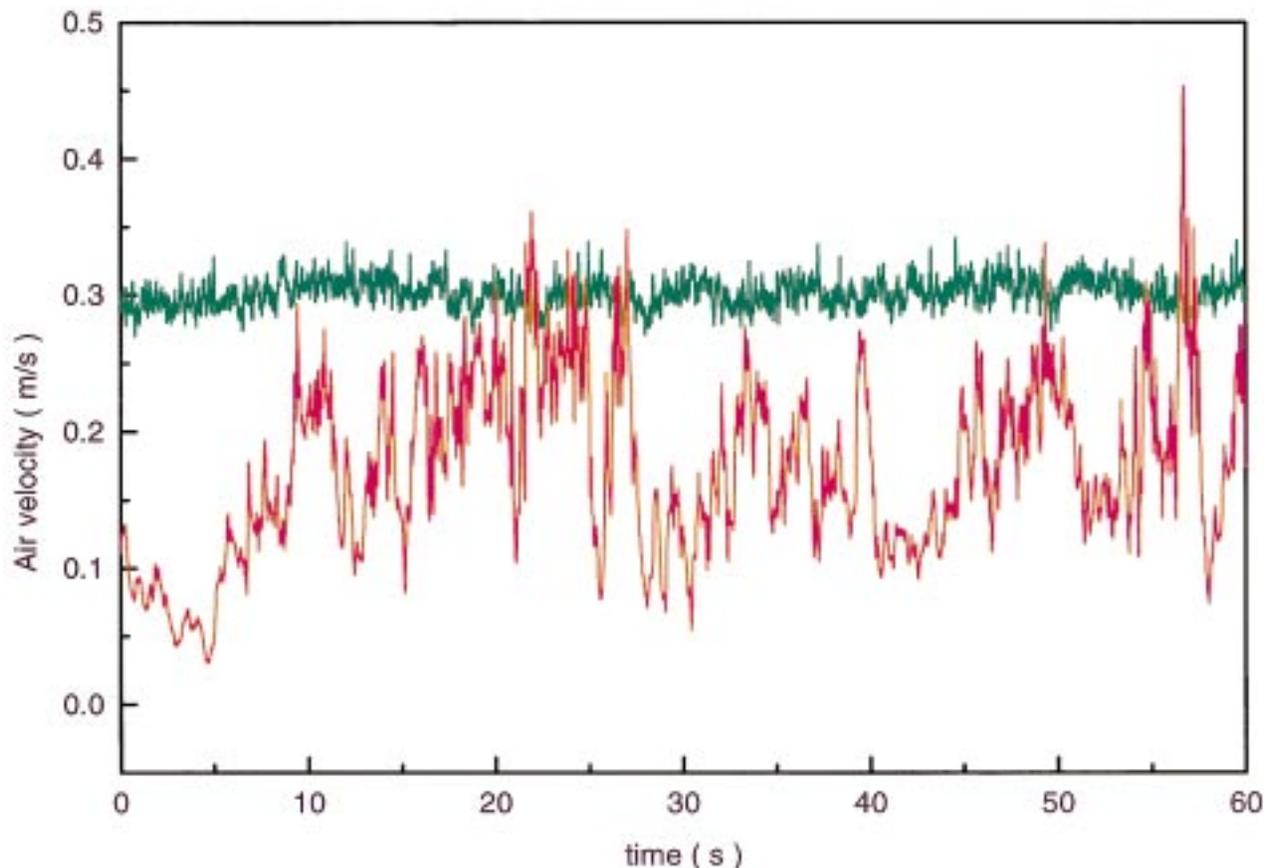


FIGURE 2. Air velocity measured over 1 min in the new chamber (upper trace) and in a 0.45 m<sup>3</sup> chamber with conventional fan (lower trace). Mean velocity and turbulence values are respectively  $0.30 \pm 0.011$  m/s (3.7%) and  $0.17 \pm 0.064$  m/s (37%).

and at five horizontal positions (see scheme in Figure 1), showed a very uniform velocity field. This is one of the most important requirements aimed at in the design of the chamber, because it allows the measurement of air velocity at a single position or using a precisely controlled motor, as we did, to avoid air velocity measurements once a calibration curve has been obtained (at least as long as the surface roughness of the test material does not change markedly). When mixing with conventional (3-blade propeller type) fans, to the contrary, the eddies generated by the fan cause wide variations of air velocity from point to point and from time to time, which forces us to measure air velocity at several points (and the choice is always difficult) and to average them over a relatively long time span. Guo et al. (18) in their recent analysis for the revision of emission testing protocols have evidenced well this point also with experimental data. Figure 2 shows the velocity fluctuations (i.e., the turbulence) in the new chamber and in a 0.45 m<sup>3</sup> chamber with a conventional fan as recorded over 1 min: from a value of 37% of the mean velocity in the latter chamber (but values in excess of 50% have been observed) turbulence is down to 3% in the new chamber. The latter value is exceeded at a few positions near the gauze or at the border of the boundary layer. The measurements in the chamber with a conventional fan have been carried out at the center of a PVC tile 45 × 50 cm at 10 mm above its surface; the tile was placed at 20 cm above the chamber bottom, and the fan was placed in front of and 30 cm above the tile, blowing toward it.

Figure 3 shows the vertical profiles obtained in the new chamber, with two different fan rotation speeds, in the central position of the controlled space (position 6) and 10 cm windward on the chamber axis (position 2). The graphs show the two boundary layers and show a good overlapping of the

profiles at the two positions, which indicates a homogeneous velocity field in the space.

The relationship between fan rotation speed (rpm =  $x$ ) and air velocity (m/s =  $y$ ) has been investigated measuring the air velocity in the center of the controlled space (position 6, 50 mm height) at different fan speeds between 300 and 750 rpm. The interpolation of the data points (correlation coefficient  $r = 0.9998$ ) is described by the linear equation:

$$y = 0.000702x - 0.123$$

The vertical velocity profiles (Figure 3) show that the boundary layer has a thickness of about 20–25 mm. This should be considered in establishing protocols for emission rate determinations whereby air velocity measurements are requested (until now it was generally requested to measure air velocity 1 cm above the surface of the emitting material).

**First Results on Material Testing. Influence of Air Velocity and Turbulence on Pure Evaporation Sources.** The emission rate of vapors from any source depends on the mass transfer from inside the source to the surrounding air. The mechanism of mass transfer may vary between two limiting conditions: (a) the resistance to mass transfer is entirely in the gas phase (boundary layer), e.g., evaporating liquid or (b) the resistance to mass transfer is entirely within the emitting material, e.g., solvent residue in some solid material. Only for the first type of sources should air velocity have a significant impact on the emission rate because it acts directly on the vapor concentration gradient in the air. In many real sources, a mixture of the two situations occurs, sometimes the former prevailing in the beginning of the emission period and the latter successively (e.g., paints).

Guo et al. (18) reported preliminary results on the emission rate of 1,4-dichlorobenzene (type (a) source), showing that

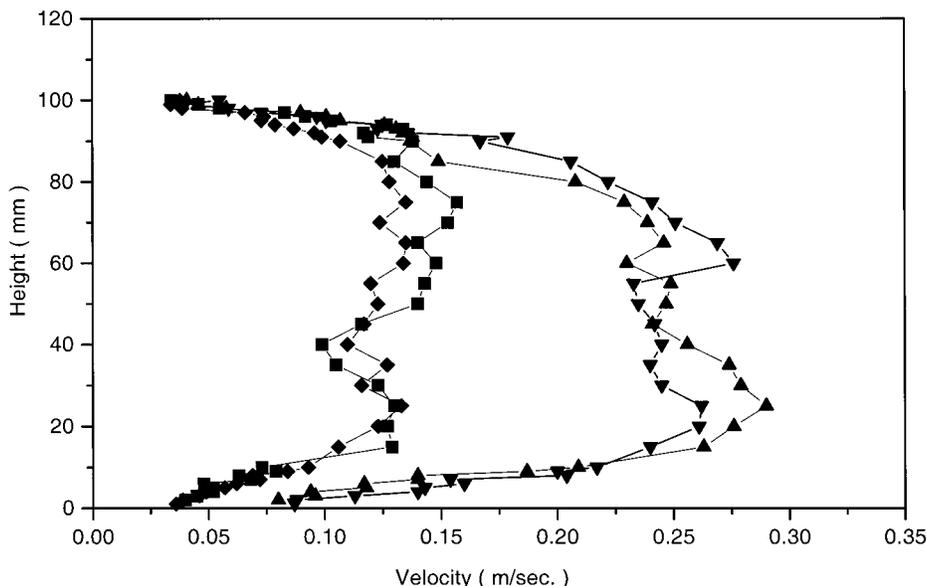


FIGURE 3. Vertical velocity profiles at two positions (nos. 2 and 6) and for two fan rotation speeds (300 and 500 rpm, respectively, squares/diamonds and triangles) in the new chamber (100 mm is the height of the test compartment, see Figure 1).

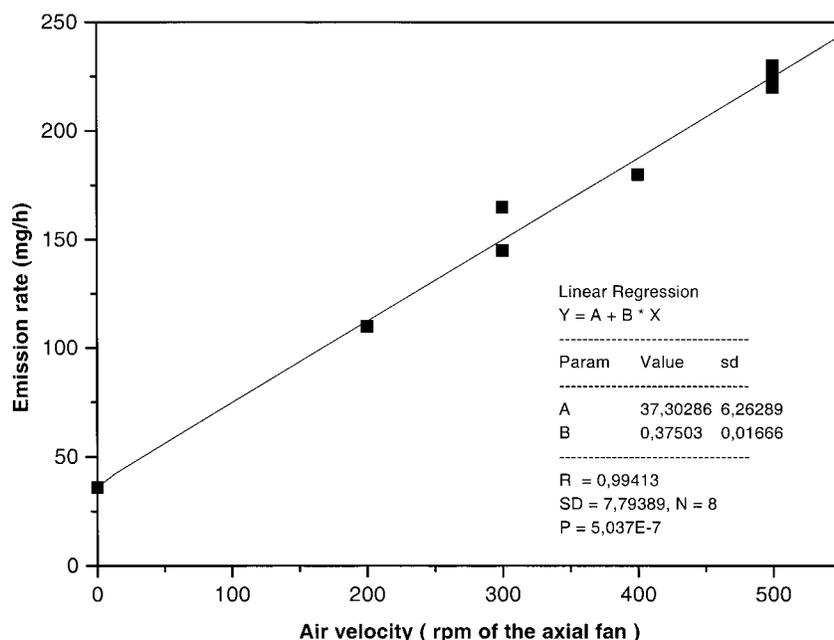


FIGURE 4. Emission rate increase as a function of air velocity for a pure 1,4-dichlorobenzene source (*A*, intercept; *B*, slope; *R*, correlation coefficient; *SD*, standard deviation of correlation; *N*, number of data points; *P*, probability, i.e., confidence level of the correlation. See the text for the relationship between rotation speed of the fan and air velocity).

in a test chamber with conventional mixing it changed considerably with fan velocity. The investigation on this type of source has been carried out, in the preliminary setup, with 1,4-dichlorobenzene crystals and with liquid *n*-decane. Both sources had the geometry of a Petri dish (57 mm i.d. or 25.5 cm<sup>2</sup>), which was accommodated in a hole in the bottom of the box; the Petri dish was directly placed onto the plate of a balance, whose reading enabled to follow the weight losses of the pure compounds. From the weight difference over a certain time, under constant air velocity and turbulence, the emission rate was determined at four fan speeds and separately with zero air velocity. The latter was determined by weight loss of a Petri dish containing the pure compound placed out of the chamber, under a beaker upside down, in a way that no vapor saturation occurred and evaporation could proceed freely. The results for dichlorobenzene are shown in Figure 4. For both compounds, the

slope of the regression line is close to 0.4 mg h<sup>-1</sup> rpm<sup>-1</sup>; considering that each 100 rpm increment corresponds approximately to 0.07 m s<sup>-1</sup> and normalizing for the evaporation area, this corresponds roughly to an increment of 0.2 mg h<sup>-1</sup> cm<sup>-2</sup> for each cm s<sup>-1</sup> increment in air velocity.

Zhang and Haghghat (14) have reported that the influence of turbulence on the evaporation rate of water appears to be small. A good condition to test this influence would be a large change in turbulence with no change in velocity. Such a situation was observed to be reasonably well approximated at two positions in the preliminary setup (nos. 2 and 10, see Figure 1), where vertical profiles have been measured. At the same fan speed, in fact, there is little difference in air velocity but about a factor 3 difference in turbulence. Therefore, an experiment was carried out in which the weight losses of 1,4-dichlorobenzene from the Petri dish have been measured at the two positions. The results are presented in Table 2.

## 1,2-propanediol from paint

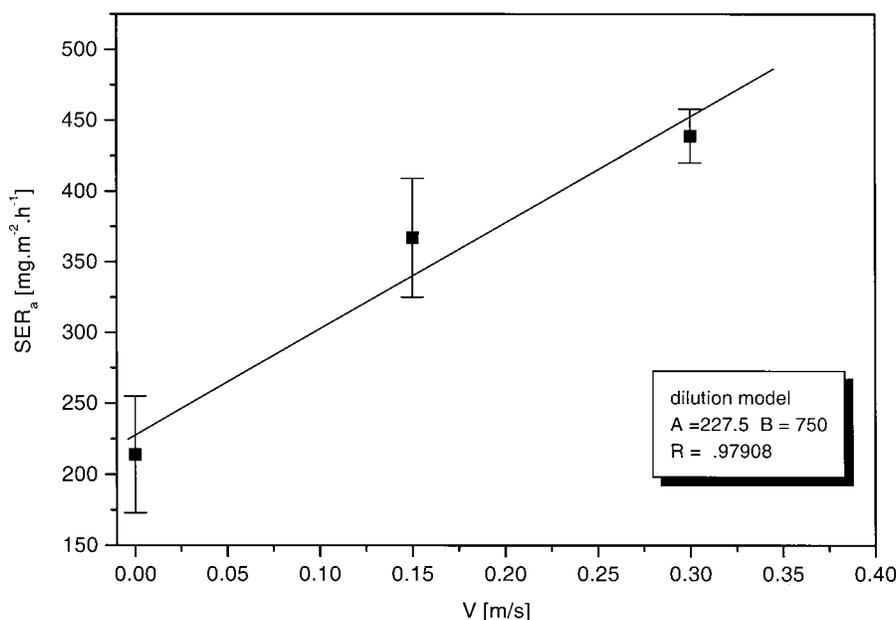


FIGURE 5. Increase of the area specific emission rate of 1,2-propanediol as a function of air velocity for a water-based latex paint (see caption of Figure 4 for the meaning of *A*, *B*, and *R*).

TABLE 2. Influence of Turbulence on the Evaporation Rate of 1,4-Dichlorobenzene (Preliminary Setup)

	position 2	position 10
mean velocity <sup>a</sup> [m s <sup>-1</sup> ]	0.272	0.245
mean turbulence <sup>a</sup> [m s <sup>-1</sup> ]	0.0501	0.0170
weight loss [g]	0.62	0.59
weight loss rate [g h <sup>-1</sup> ]	0.21	0.20

<sup>a</sup> Above the boundary layer.

The evaporation rates observed at the two positions show a difference of 10 mg h<sup>-1</sup> (5%), which is compatible with the small difference in air velocity. This leads to the conclusion that, under the experimental conditions adopted and for the turbulence interval tested, the influence of turbulence on the evaporation rate, if any, is very small. It should be noted that, in the preliminary setup, the PTFE grid caused considerably more turbulence than the gauze in the final chamber. The result of this preliminary test should be confirmed by further investigations on more emitting materials and in a wider turbulence range (e.g., changing the mesh size of the gauze filtering the eddies of the recirculating air).

**Influence of Air Velocity on Sources Limited by Internal Diffusion.** To test the influence of air velocity on VOC emission from a solid material where mass transfer can be assumed to be limited by internal diffusion, we tested a new type of PVC tile (vinyl cushion, 50 × 50 cm) that is a thick (6.2 mm) composite requiring no glue for laying. The principal VOCs emitted from a specimen 25 × 32 cm, with edges and bottom covered by self-adhesive aluminum sheet, were determined under conditions of steady-state concentration with velocities of ~0.08 and 0.20 m s<sup>-1</sup>, respectively. The specimen was placed on the bottom of the chamber. The chamber background was controlled and found to be about 30 and 10 μg m<sup>-3</sup> for the total VOCs and the highest peak, respectively (toluene equivalents). These values include the background emission of Tenax samplers, which contributes about half. The smallest concentrations of single compounds measured in the chamber are at least 10 times the background concentrations. Sampling and chemical analyses were carried

TABLE 3. Emission Rate (μg h<sup>-1</sup>) of a PVC Tile at Different Air Velocities

compound	~0.08 m s <sup>-1</sup>	0.20 m s <sup>-1</sup>	% difference
2-butanone	174	166	-4.6
toluene	15.9	15.3	-3.8
2-butoxyethanol	113	109	-3.5
phenol	201	210	+4.5
1-methyl-2-pyrrolidone	18.7	18.6	-0.5

out in triplicate, and the associated coefficients of variation were below 3.5%. The results are reported in Table 3 in terms of average emission rate.

All the compounds, except phenol, show a tendency to decrease from the first to the second set of measurements; this is due to the decrease of the emission rate over time, known from previous measurements. The only compound showing an increase is phenol, and the increase is significant at the 0.05 confidence level; this result seems to indicate an influence, though small, of air velocity on the emission rate of this compound. To explain this fact, we made the hypothesis that phenol was concentrated in the very first layer of the material and that, within this layer, diffusion was not much slower than transport within the gas phase. The composite tile investigated, in fact, has a first 1 mm layer of pure PVC. Measurements of the emission rate of the back surface of the tile showed a 3-fold lower emission rate of phenol, a fact compatible with the above-mentioned hypothesis.

The results of these tests confirm the theoretical expectation that, for this type of sources, air velocity has a very small impact on the emission rate; however, they show that measurable differences in the emission rate may occur at different air velocities.

**Influence of Air Velocity on the Emission Rate of a Water-Based Paint.** Paints are sources with an intermediate character between the two source types described above: in fact, they may be considered like pure evaporative sources in a first phase after preparation of the paint film, and later they become more similar to sources limited by internal diffusion. Tests have been carried out in the frame of the VOCEM project

on the influence of air velocity on the emission rate of paint specimens, measuring test chamber concentrations of target compounds at three air velocities (0.0, 0.15, and 0.30 m/s). Paint specimens of 9 × 25 cm were prepared manually on stainless steel supports using a film applicator with 0.20 mm recess height. The preparation of the specimens overall requested less than 10 min. VOC sampling from the chamber on Tenax samplers was carried out at different times after  $t_0$  (1, 3, 6, 24, 48, and 72 h;  $t_0$ , time of introduction of the paint film into the chamber) for a duration of about 5 min. Area-specific emission rate values at time zero were derived from the concentration values through a mathematical model (dilution model, see ref 19). To offset the variability due to the film preparation, three tests were carried out at each air velocity. The mean results obtained at each velocity for 1,2-propanediol, the main compound emitted from the water-based paint selected for these tests, are represented in Figure 5. The error bars ( $\pm 1$  SD) associated with each mean value of the specific emission rate indicate the fluctuations of the whole procedure, i.e., the preparation of the paint specimens and the chemical determination of the concentration. The linear correlation between air velocity and emission rate shows a ~30% increase of the latter doubling air velocity; this means that air velocity in the test chamber must be carefully controlled to obtain coherent data for the emission rate.

The investigations reported above, which lead to the quantification of the dependence of emission rate on air velocity for few materials, are only initial; they should be pursued testing other "wet" or liquid materials, like waxes, glues, etc.

**Adsorption Properties of the Chamber.** The adsorption (sink) properties of the new chamber are being studied with some target compounds, and the results will be reported elsewhere. Preliminary results show that adsorption is low, i.e., recovery is high, for high boiling compounds even at very low concentrations (85% for TXIB at  $\sim 20 \mu\text{g m}^{-3}$ ). Investigations on the dependence of the recovery on air change rate are ongoing.

## Acknowledgments

The project VOCEM (Further Development and Validation of Test Chamber Method for Measuring VOC Emissions from Building Materials and Products) is supported by the Standard, Measurement and Testing Programme of the European Commission (Contract SMT4-CT95-2039). The partners, in addition to the JRC-EI, are the following: BYK-Chemie GmbH, Wesel (Germany); Centre Scientific et Technique du Bâtiment, Marne-la-Vallée (France), coordinator; Sommer SA, Wiltz (Luxembourg); Tikkurila OY, Vanta (Finland); TNO Building and Construction Research, Delft (The Netherlands); Vorwerk & Co, Hameln (Germany) in association with Teppich Forschung International, Aachen (Germany); VTT Technical Research Centre of Finland, Espoo (Finland). The contributions by Herbert Schauenburg for the GC-MS identification of the emitted compounds and the adsorption experiments and by Stylianos Kephelopoulos for the model calculations of paint emissions are gratefully acknowledged.

## Supporting Information Available

Six figures and two tables providing more detailed information (8 pages). This material is available free of charge via the Internet at <http://pubs.acs.org>.

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Received for review April 7, 1998. Revised manuscript received February 8, 1999. Accepted February 15, 1999.

ES980346Y